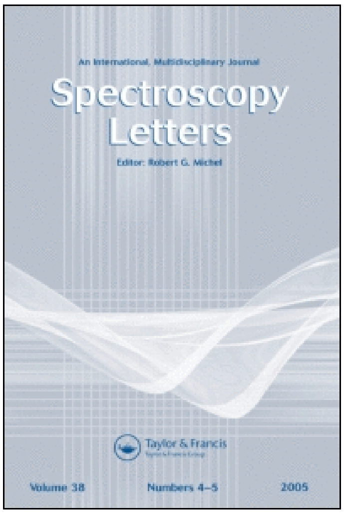

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Joseph Sneddon[a]; Charles O. Noble[a]; Mark V. Smith[a]; James N. Beck[b]
[a] Department of Chemistry, McNeese State University, Lake Charles, Louisiana, USA [b] Department of Physical Sciences, Nicholls State University, Thibodeaux, Louisiana, USA

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Simultaneous Determination of Cadmium, Chromium, Lead, and Manganese in Smoke from Burning Oil by Impaction Graphite Furnace Atomic Absorption Spectrometry

Joseph Sneddon,[1],* Charles O. Noble,[1] Mark V. Smith,[1] and James N. Beck[2]

[1]Department of Chemistry, McNeese State University, Lake Charles, Louisiana, USA
[2]Department of Physical Sciences, Nicholls State University, Thibodeaux, Louisiana, USA

ABSTRACT

Impaction-graphite furnace atomic absorption spectrometry (I-GFAAS) has been used to directly and simultaneously determine cadmium, chromium, lead, and manganese in the smoke or plume from burning crude oil in laboratory-controlled conditions. Concentrations in the low ng/m^3 can be determined for all four metals. Cadmium and lead appear

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*Correspondence: Joseph Sneddon, Department of Chemistry, McNeese State University, Lake Charles, LA 70609, USA; E-mail: jsneddon@mail.mcneese.edu.

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in the smoke in the first 30 sec of the burn whereas up to 120 sec is required for all the chromium and manganese to be found in the smoke.

Key Words: Simultaneous impaction-graphite furnace atomic absorption spectrometry; Burning oil; Smoke; Cadmium; Chromium; Lead; Manganese.

INTRODUCTION

Impaction-graphite furnace atomic absorption spectrometry (I-GFAAS) is a useful technique for the direct and near real-time determination of metals in aerosols (a solid or liquid in a gaseous medium).[1] The basic principle involves an aerosol being drawn by vacuum at a fixed flow rate and known time through an impactor. The impactor exit is positioned at the entrance port of a graphite furnace. After sampling, the graphite furnace is then atomized for quantitative metal determination by conventional graphite furnace atomic absorption spectrometry. The basic principles, design, description, and performance of I-GFAAS is described in detail elsewhere.[1] The system has been applied to the determination of selected metals in a clean room,[2] cigarette smoke,[3] and laboratory air.[4] Other techniques have potential for such measurements including electrostatic precipitation atomic absorption spectrometry[5,6] but require a more complex system plus concerns over collection efficiency.

Recent work from this laboratory has focused on the fate of selected metals in burning oil.[7–10] This involves the determination of metals in unburned oil, the oil then being burned, and then the residue determined for the same metals by flame atomic absorption spectrometry. Burning of oil has been proposed as a form of remediation for large (>10,000 gallons) oil spills.

This work describes the use of simultaneous I-GFAAS to directly determine selected metals in the smoke or plume from burning Venezuelan crude oil under controlled laboratory conditions.

EXPERIMENTATION

Instrumentation

A detailed description of the I-GFAAS system is described elsewhere.[7] A Smith-Hieftje 8000 automatic atomic absorption spectrometer (Thermo Jarrell Ash-Baird, now called ThermoElemental, Franklin, MA) equipped with CTF 188 controlled temperature atomizer was used. The system was used in the simultaneous mode and determined the four metals (cadmium, chromium, lead, and manganese).[3] Experimental and instrumental conditions for the simultaneous determination of the four metals are shown in Table 1. This involved a compromise in conditions due to the different properties of the four metals. The maximum ashing or pyrolysis step was 250°C to prevent loss of cadmium and an atomization step of 2350°C was required to atomize chromium. The oil was burned in a burning chamber.[8] This was constructed using 7-mm thick Plexiglas. The chamber measured 50-cm × 20-cm × 20-cm with a hinged top. Four 13-mm air intake ports were drilled on each side, approximately 3-cm from the bottom. The burning smoke/air was drawn through a 7-mm diameter Tygon tube connected directly to the impactor-system[3] by a 0.5-hp ALATEC vacuum pump (Franklin Electronics, Bluffton, IN).

Reagents

All chemicals used were reagent grade. Standards were made from 1000 μg/mL Metallo Organic standards in mineral oil obtained from High Purity Standards (Charleston, SC). Venezuelan crude oil was obtained from

Table 1. (a) Experimental and (b) instrumental conditions for the simultaneous determination of the four metals in burned crude oil smoke by I-GFAAS.

Step number	Process	Temperature (°C)	Ramp time (sec)	Hold time (sec)
(a) Experimental				
1	Injection/dry	150	2	3
2	Pyrolysis 1	200	15	5
3	Pyrolysis 2	250	15	5
4	Atomize	2,350	0	6
5	Clean	2,350	—	2
	Cadmium	Chromium	Lead	Manganese
(b) Instrumental				
Wavelength (nm)	228.8	357.9	217.0	279.5
Lamp current (mA)	4.0	4.0	4.0	4.0
Bandpass (nm)	0.40	0.40	0.40	0.40
Measurement (absorbance)	Peak area	Peak area	Peak area	Peak area
Background correction	Smith-Hieftje	Smith-Hieftje	Smith-Hieftje	Smith-Hieftje
Purge gas	Argon	Argon	Argon	Argon

a local refinery (Westlake, LA). The crude oil was a random sample taken from a tanker and stored in a five-gallon acid rinsed/deionized water plastic container. Methyl isobutyl ketone (MIBK) was used as the solvent for dilution and removal of oil.

Procedure

A mass of crude oil, typically 3-mL, was accurately weighed into a contamination-free (of cadmium, chromium, lead, and manganese) aluminum container. The concentrations of cadmium, chromium, lead, and manganese in crude oil was simultaneously determined using standard simultaneous GFAAS methods. The oil was then burned, typically 30–150 sec from ignition to extinction. During this time, the impactor sampled the air/burning smoke at a flow rate of 5 L/min. After extinguishing the burning oil, the impactor could be closed or continue to sample the air.

Accuracy of I-GFAAS for direct and simultaneous determination of metals has been established previously.[1]

RESULTS AND DISCUSSION

Simultaneous Determination of the Four Metals in Crude Oil/Smoke from Burning Crude Oil and After Extinguishing the Burning Crude Oil

The four metals chosen for this study are found in Venezuelan crude oil in the low μg/mL levels.[11] Quantifying the absorbance signals from the four metals in the burning smoke was achieved by comparison to aqueous standards as described previously (albeit for cigarette smoke but the same principal).[3] Detection limits (signal-to-noise ratio of three) for the three metals (chromium, lead, and manganese) were in the low ng/m^3 with cadmium in the sub-ng/m^3.[1]

Concentrations of the four metals in smoke are shown in Table 2. These results are the mean of three trials with a precision for each of the four results of less than 10%. These results were obtained by burning 3 mL of the oil for a set time such as 30 sec with simultaneous collection on the impaction system. After burning/collection for the selected time; the impaction system was closed for subsequent measurement. A further 3 mL of crude oil was added and then burned and the smoke collected for 60 sec. This was repeated at 30 sec intervals up to approximately 150 sec. At this time the crude oil flame was extinguished, presumably because all the flammable material in

Table 2. Concentrations of cadmium, chromium, lead, and manganese in burning smoke from Venezuelan crude oil collected on the impaction-graphite furnace system and subsequently quantified by simultaneous multielement atomic absorption spectrometry.

	Concentration (ng/m^3)				
Metal	After 30 sec	After 60 sec	After 90 sec	After 120 sec	After 150 sec
Cadmium	2	2	2	2	2
Chromium	16	17	19	29	29
Lead	8	9	10	9	9
Manganese	11	11	10	15	16

the crude oil was turned into smoke. For the four metals investigated the concentrations were in the low ng/m^3. For lead and cadmium the metal entered the smoke in the first 30 sec whereas for manganese and chromium it took 120–150 sec. Cadmium and lead concentrations remained (essentially) constant throughout the five time trials investigated. This suggests that all (volatile) cadmium and lead was removed in the first 30 sec. The remaining four time trials did not find any change in cadmium or lead. This could be related to the volatility of the metals. It is quite possible that the metals would be present in various chemical forms in the crude oil: one chemical form more volatile than the other chemical form. The longer the flame burned the higher the temperature. However, it was noted that even after 30 sec some manganese and chromium was in the smoke.

After burning at the selected time, the remaining oil was removed by MIBK and determined for the four metals by conventional GFAAS. After 30 sec, no cadmium and lead was found although an absorbance signal (not quantified) was found for chromium and manganese. After 150 sec, no absorbance signal (again not quantified) was found for any of the four metals. These results support the previous hypothesis that the volatility of the metal will determine when it is removed from the burning oil.[9]

Several hours after the burning had been completed, an analysis of the air for the four metals of interest showed absorbance signals.

CONCLUSION

This work has continued to show that the use of GFAAS can be a valuable tool for determining metals in aerosols; in this work, smoke from burning crude oil. Due to the concern over standards, the results must be regarded as somewhat semi-quantitative. Nevertheless, the results do provide some insight as to the fate of selected metals in burning crude oil.

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